DEPARTMENT OF
ECOLOGY
State of Washington

Standard Operating Procedure EAP108, Version 1.10

Collecting In Situ Water Quality Data

February 2019
Publication 19-03-206

Purpose of this document

The Washington State Department of Ecology develops Standard Operating Procedures (SOPs) to document agency practices related to sampling, field and laboratory analysis, and other aspects of the agency's technical operations.

Publication Information

This SOP is available on the Department of Ecology's website at
https://fortress.wa.gov/ecy/publications/SummaryPages/1903206.html.

Ecology's Activity Tracker Code for this SOP is 12-074.

Recommended citation:
Wolfe, J. 2019. Standard Operating Procedure EAP108, Version 1.10: Collecting In Situ Water Quality Data. Washington State Department of Ecology, Olympia.
https://fortress.wa.gov/ecy/publications/SummaryPages/1903206.html.

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Standard Operating Procedures for Collecting In Situ Water Quality Data

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Although Ecology follows the SOP in most instances, there may be instances in which the Ecology uses an alternative methodology, procedure, or process.

SOP Revision History

Revision Date	Rev number	Summary of changes	Sections	Reviser(s)
1/4/17	1.1	Added Footers, updated references	All	Meghan Rosewood-Thurman
1/10/17	1.2	Updated glossary, general edits	All	Meghan Rosewood-Thurman
1/20/17	1.3	Updated Glossary, general edits	All	Meghan Rosewood-Thurman
1/26/17	1.4	Updated References, general edits	All	Meghan Rosewood-Thurman
1/30/17	1.5	3.10 removed bold 3.11 replaced PSAT definition 3.13 spelling of Protocol 3.16 re-wrote definition for µS/cm 5.4 Labeled soaker bottle in Fig 2 6.0 edited cites 6.1.1.2 reference Figure 2 6.3.1.1 Added Figure 4 and ref. to it 6.3.1.2 ref. to Figure 4 6.3 Renumbered Fig to 5; referenced it 10.13 Added cite: WOW. 2004	3.0 5.0 6.0 10.0	Glenn Merritt
2/13/17	1.6	Added Reviewers	Signature Page	Meghan Rosewood-Thurman
2/16/17	1.7	Removed drafted dates	Signature Page	Meghan Rosewood-Thurman
12/11/17	1.8	Replaced Tin Foil with User Manual Changed frequency of calibration Changed frequency of calibration Changed frequency of calibration Removed BOD procedure Changed frequency of probe check	5.13 6.1.1 6.1.2 6.1.3 6.1.3.1 6.5	Brian Engeness
12/21/17	1.9	Added new citation of SOP EAP127 Referenced citation of EAP127 Removed sentence	10.8 6.1.3 6.1.3.1	Brian Engeness
12/28/17	1.10	Updated Footers, general edits	All	Meghan Rosewood-Thurman
8/7/18	1.10	Minor edits and format changes for recertification	All	Tom Gries

1.0 Purpose and Scope

1.1 This document is the Environmental Assessment Program's (EAP) Standard Operating Procedure (SOP) for measuring in situ water quality in rivers and streams for the Watershed Health Monitoring (WHM) program. It includes procedures for both of the WHM protocols. The Narrow Protocol is typically accomplished by wading upstream. The Wide Protocol is typically accomplished by floating on rafts. This SOP is also used in the Ambient Biological Monitoring Program.

1.2 In situ water quality is measured with a multi-parameter probe and includes: temperature, specific conductivity, pH, dissolved oxygen (DO) and oxygen percent saturation (PSAT).

2.0 Applicability

2.1 This SOP contributes to both of the WHM protocols: Narrow and Wide.

2.2 This SOP is used in conjunction with several others to complete a data collection event (DCE) for the WHM program.

3.0 Definitions

3.1 DCE: The Data Collection Event is the sampling event for the given protocol. Data for a DCE are indexed using a code which includes the site ID followed by the year, month, day, and the time (military) for the start time of the sampling event. For example: WAM06600-000222-DCE-YYYY-MMDD-HH:MM. One DCE should be completed within one working day, lasting 4-6 hours, on average.

3.2 DI: Deionized water.

3.3 DO: Dissolved Oxygen. The concentration of dissolved oxygen in a water sample. Reported in mg/L.

3.4 EAP: Environmental Assessment Program

3.5 Ecology: The Washington State Department of Ecology

3.6 Index station: Index station: The distinct point location mapped by the site coordinates obtained from the Washington Master Sample List. The index station is called "X" and is generally located at major transect F; however the point may occur at any elevation in the stream between transects A and K.

3.7 LDO: Luminescent dissolved oxygen; dissolved oxygen values are measured by pulses of LED light.

3.8 Lotic: Flowing water systems such as streams and rivers.

3.9 Narrow protocol: The set of Watershed Health Monitoring SOPs that describe data collection at wadeable sites with an average bankfull width of less than 25 m at the index station.

3.10 pH: a measure of hydrogen ion concentration; a measure of the acidity or alkalinity of a solution. Aqueous solutions at 25°C with a pH less than seven are acidic, while those with a pH greater than seven are basic or alkaline.

3.11 PSAT (% sat): Percent saturation of oxygen is calculated as the percentage of dissolved oxygen relative to that concentration which occurs when completely saturated at the ambient temperature, pressure, and salinity (WOW 2004).

3.12 QAMP: Quality Assurance Monitoring Plan. The QAMP for WHM is Cusimano et al. (2006). An updated version is in early stages of development.

3.13 Site: A site is defined by the coordinates provided to a sampling crew and the boundaries established by the protocol's site layout method (Hartman, 2017 (SOP EAP105) for the Wide Protocol; Merritt, 2017 (SOP EAP106) for the Narrow Protocol). Typically, a site is centered on the index station and equal in length to 20 times the average of 5 bankfull width measurements. Sites cannot be longer than 2 km nor shorter than 150 m. Narrow protocol sites range from 150 m to 500 m long. Wide Protocol sites are at least 500 m long and up to 2 km long. The most downstream end of a site coincides with major transect A; the most upstream end coincides with major transect K.

3.14 Specific Conductivity: Electrical conductivity is a measure of water's ability to conduct electricity, and therefore a measure of ionic activity and content. It is the reciprocal of specific resistivity. Specific conductivity is conductivity adjusted to 25˚ C (reported in µS/cm at 25˚ C). This is what most field conductivity meters report.

3.15 Thalweg station or transect: One of one hundred (100) equidistant measurement locations in the thalweg, across the length of a site. For example the thalweg stations at/above each major transect are named as follows:

- A0, A1, A2, A3, A4, A5, A6, A7, A8, A9,
- B0. B1, B2, B3, B4, B5, B6, B7, B8, B9,
- C0, C1, C2, C3, C4, C5, C6, C7, C8, C9,
- …
- J0, J1, J2, J3, J4, J5, J6, J7, J8, J9, and
- K0.

3.16 µS/cm: micro-Siemens per centimeter, the unit that we use for measurement of electric conductance.

3.17 WHM: Watershed Health Monitoring, a status and trends monitoring program within the Environmental Assessment Program at the Washington State Department of Ecology.

3.18 Wide protocol: The set of WHM SOPs that describes the sample and data collection at non-wadeable sites or sites wider than 25 m bankfull width. It is an abbreviated version of the Narrow Protocol and is typically accomplished by use of rafts.

4.0 Personnel Qualifications/Responsibilities

4.1 This SOP pertains to all Natural Resource Scientists, Environmental Specialists, Interns and Technicians in Ecology's EA Program, as well as any other qualified staff collecting and entering data for WHM.

4.2 This method is performed by 1 or more persons, at every site, at the beginning and end of the DCE. Daily quality control (QC) checks precede and follow sampling. Other QC tasks are required less frequently than daily. Staff performing this method must have been trained.

4.3 All field staff must comply with the requirements of the EA Safety Manual (Ecology, 2017). Have a full working knowledge of the procedures in Chapter 1 'General Field Work,' especially the sections 'Working in Rivers and Streams,' and 'Fall Protection'. When sampling from a boat, one person onboard must be a qualified boat operator and all persons onboard must be familiar with Chapter 3 of the EA Safety Manual, 'Boating.'

4.4 All field staff must have completed the annual WHM program field training and be familiar with the set of SOPs, that when combined, describe a full DCE for the WHM program.

4.5 All field staff must be familiar with the WHM electronic data recording method described in SOP EAP 125 (Janisch, 2017).

4.6 The field lead directing sample collection must be knowledgeable of all aspects of the project's Quality Assurance Monitoring Plan (QAMP) to ensure that credible and useable data are collected. All field staff should be briefed by the field lead or project manager on the sampling goals and objectives prior to arriving to the site.

4.7 All field staff must comply with SOP EAP070 (Parsons et al., 2016) 'Minimizing the Spread of Invasive Species'.

5.0 Equipment, Reagents, and Supplies

5.1 Field tablet, electronic field forms

5.2 HQ40d Calibration Form (Appendix)

5.3 HQ40d Portable Multi-Parameter Meter (Figure 1)

5.4 PHC28101 IntelliCAL pH Ultra Electrode (Figure 2)

5.5 LDO101 IntelliCAL Standard Dissolved Oxygen Probe (Figure 2)

5.6 CDC401 IntelliCAL Standard Conductivity Probe (Figure 2)

5.7 Hach "Singlet" Single-Use pH Buffers; 4.01, 7.00, 10.01

5.8 Thermo Scientific Orion Pure Water pH Buffer; 6.97

5.9 Hach IntelliCAL™ 2.44M KCl PHC281 filling solution for pH probe

5.10 Ricca Chemical Conductivity/TDS Standard; 100 µmho/cm

5.11 De-ionized water (DI) to rinse equipment

5.12 Lab tissues (e.g., Kim-wipes®)

5.13 Hach HQ40d Multi-Parameter User Manual

5.14 4 AA batteries

5.15 500ml plastic beaker

5.16 DO grab sample (Winkler) supplies

5.17 Funnel tube surface sampler

5.18 BOD bottle with glass stopper and plastic cap

5.19 Manganous sulfate monohydrate reagent bottle with 2 mL disposable transfer pipette

5.20 Alkali-iodine-azide reagent bottle with 2 mL disposable transfer pipette

5.21 Access gear (boats, or waders and boots). This should be pre-cleaned to avoid the spread of invasive species. See SOP EAP070 (Parsons et al., 2016) for more information.

Figure 1. HQ40d Multi-Parameter Meter

Figure 2. PHC28101 IntelliCAL pH Ultra Electrode, CDC401 IntelliCAL Standard Conductivity Probe, and LDO101 IntelliCAL Standard Dissolved Oxygen Probe.

6.0 Summary of Procedure

The following methods were derived, in part, from Status and Trends Monitoring for Watershed Health and Salmon Recovery. Draft Field Data Collection Protocol: Narrow Streams (Merritt and Hartman 2013) and Collection, Processing, and Analysis of Stream Samples (Ward 2012).

6.1 Meter Calibration and Pre-Sampling Calibration Accuracy Check

6.1.1 **Calibrate the pH electrode** at the beginning of the work week and after every two or three days if using the probe daily. Each time calibrating, use new packets of the color coded Hach single use calibration buffers. Ensure that the temperatures of the buffers are 15˚ C or higher (but not above 30˚C). Conduct a 3-point calibration with pH 4.01, 7.00, and 10.01 calibration buffers according to instructions provided in the user's manual (Hach, 2010a).

6.1.1.1 Ensure that the pH electrode is full of IntelliCAL™ 2.44M KCl PHC281 solution. If needed, top off the probe fill chamber with filling solution (Figure 3).

Figure 3. Close-up of electrolyte filling hole on pH probe

6.1.1.2 Before removing the electrode from the soaker bottle (Figure 2), unscrew the bottle base from the bottle cap. This will ensure that electrolyte is not suctioned out of the probe. Remove the bottle cap and put the bottle aside where it will not spill or become contaminated.

6.1.1.3 Thoroughly rinse the electrode with DI water prior to calibration and in between buffers. Be sure to shake off excess water.

6.1.1.4 Let the pH electrode equilibrate in each calibration buffer for at least one minute before taking a reading. Ensure the electrolyte filling-hole is open for an accurate measurement.

6.1.1.5 Stir the electrode gently during calibration. Do not rest the electrode on the bottom or sides of the container.

6.1.1.6 After calibrating, measure the pH QC 7 buffer and compare the result to the true value of the buffer based on the buffer temperature. Record the buffer true value and measured value on the Calibration Form (Appendix). If the measured value is within 0.10 units, then proceed with sampling, if not then recalibrate and try again. Perform this accuracy check before and after the DCE.

6.1.1.7 Occasionally, clogs may form in the filling solution for the PHC281 pH probe. When this happens, it can cause inaccurate and unstable pH readings. It may be necessary to clear the pH reference junction in the tip of the probe using the following procedure:

6.1.1.7.1 Attach the probe soaker bottle to the tip of the probe and seal the cap.

6.1.1.7.2 Unplug the probe fill hole and pull the probe soaker bottle with slight pressure to suction at least ½ inch of the filling solution out of the probe.

6.1.1.7.3 Refill the probe through the fill hole.

6.1.1.7.4 Repeat this process if you find additional clogs. If clogs continue, it may be necessary to replace all the filling solution in the probe with fresh filling solution.

6.1.2 **Calibrate the conductivity probe** using 100 µS/cm Ricca Chemical Conductivity/TDS buffer. Follow the calibration instructions provided in the user's manual (Hach, 2010c). Perform calibration at the beginning of the work week before the site visit and after every two or three days if the probe is being used daily.

6.1.2.1 Use DI water and a cotton swab to scrub the contacts inside the tip of the probe.

6.1.2.2 Thoroughly rinse the probe with DI water and shake off excess prior to calibration.

6.1.2.3 After calibrating, measure the conductivity buffer and compare the result to the known value. Record this measurement on the Calibration form (Appendix). If the conductivity measurement is not within 10 µS/cm, then recalibrate. Perform this accuracy check before and after the DCE

6.1.3 **Calibrate the LDO probe** at the beginning of the work week before the site visit and after every two or three days if the probe is being used daily. Follow the calibration instructions as outlined in the Environmental Assessment Program's SOP EAP127 (Hoselton, 2018).

6.1.3.1 Do not calibrate in direct sunlight since this can cause temperature fluctuations, which will hinder the calibration process.

6.2 General Considerations and Cautions

6.2.1 Never compromise your personal safety or that of field partners. Always plan ahead to avoid falling and drowning hazards.

6.2.2 In situ measurements should be one of the first tasks you complete. It is also one of the last tasks at a DCE (the second set of measurements). Record time (military) and location (thalweg transect). Measurements should *always* be taken within the boundaries of the site (between transects A0 and K0) at the beginning and end of the data collection event. When rafting, collect the first sample near the top of the site (upstream) and collect the end sample from near the bottom of the site (downstream). When wading collect start and end samples from the same station as each other, normally the index station.

6.2.3 Choose a sample location that is representative of the site. This location should be relatively deep and non-turbulent. If possible, sample near the thalweg or predominant downstream current. Avoid back eddies and side channels.

6.2.4 To avoid sample contamination, measure parameters before other crew members enter the stream and make sure not to disturb sediment from the stream bed.

6.2.5 If sampling on foot, face upstream while obtaining each in situ measurement.

6.2.6 If sampling from a boat, avoid gas and oil contamination. Measure from near the bow while the boat is pointed upstream.

6.3 Measurements

6.3.1 Measure temperature, conductivity, pH and dissolved oxygen (mg/L, PSAT) at the beginning of the DCE.

6.3.1.1 Thermally equilibrate the pH electrode. Collect a sample of stream water with the 500 mL beaker (Figure 4) and place it in a shallow, calm, edge section of the stream. Unplug the pH electrode fill hole and carefully remove the pH electrode soaker bottle. Place the pH electrode upright in the beaker and let it sit for 3-5 minutes. Be sure that you do not submerge the pH electrode past the electrode filling solution hole.

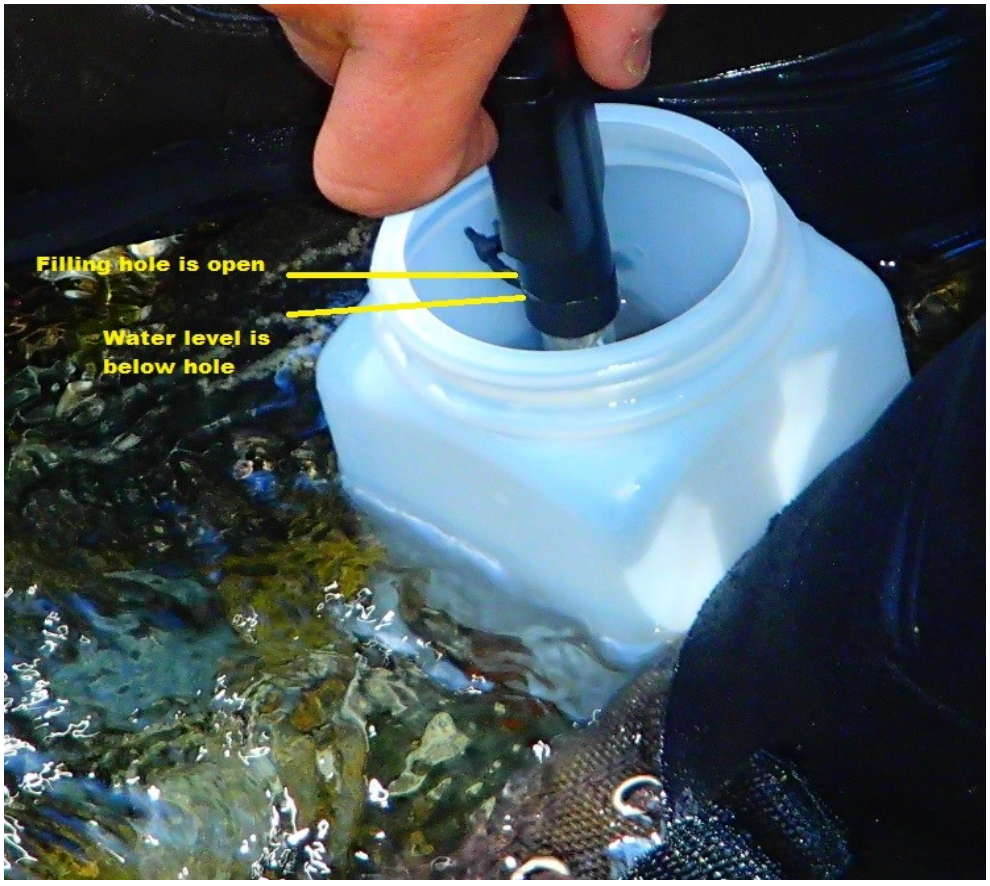

Figure 4. Measuring pH at equilibrated temperature and outside of streamflow.

6.3.1.2 Protect the pH electrode from flow-induced error. Measuring pH in flowing water can be problematic so do not place the electrode directly into the stream. Instead, re-fill the beaker with fresh, well-mixed stream water and measure from that (Figure 4). Keep the filled beaker partly submerged in stream water while taking the measurement in order to measure close to ambient stream temperature.

6.3.1.3 Measure pH in the contained water. Gently stir the pH sample with the pH electrode for several seconds while obtaining a stable sample measurement. Repeat this process until consecutive stable readings are within 0.02 pH units and the millivolts (Mv) readings are within 0.1 Mv of each other. On the field tablet, navigate to the *Chemistry Page.* Select "Get Time" button to record the time of the in situ measurements. Record the station ID (nearest transect) and record pH to the nearest hundredth (Figure 3).

6.3.1.4 Once you have recorded a stable stream pH value (Figure 5), plug the fill hole, rinse the pH probe with DI water and replace it in the soaker bottle. Make sure there is enough clean filling solution in the soaker bottle to cover the pH bulb (about ½ full). Detach the pH probe and connect the LDO and conductivity probes.

6.3.1.5 Thermally equilibrate the LDO and conductivity probes. Find a spot in the stream where the water is well mixed but not overly turbulent. Hold the LDO and conductivity probes so that they are just below the surface of the water, and completely immersed. Let them sit for 3-5 minutes.

6.3.1.6 Measure four parameters in flowing water. On the *Chemistry* sampling page (Figure 5), record temperature (˚ C, nearest tenth), specific conductivity (µS/cm at 25˚ C, nearest tenth), dissolved oxygen (mg/L, nearest tenth), and oxygen percent saturation (nearest tenth). Temperature should be measured using the LDO probe (for consistency).

6.3.2 At the end of the DCE, re-measure temperature, specific conductivity, pH and dissolved oxygen (mg/L, PSAT). Record these values (Figure 5).

6.3.2.1 Repeat steps 6.3.1.1 through 6.3.1.6.

6.4 <u>Pre and Post-Sampling Accuracy Check</u>

6.5 Before starting the DCE and after completing the DCE, recheck the accuracy of the Hach meter and electrodes. Follow the calibration accuracy check procedures outlined in section 6.1.Verify that the buffers fall within the specified ranges: pH within 0.1 pH units, conductivity within 10µS/cm, DO water saturated air between 97.5% and 102.5%.

6.5.1 If the measured value of the QC solution is within the specified range then record this information on the calibration form. If it is outside the specified range then recalibrate **and resample** if possible. If the in situ pH or conductivity measurement does not meet the QC criteria, then collect and ship a water sample for the failing measurement as outlined in SOP EAP095 (Hartman, 2017)

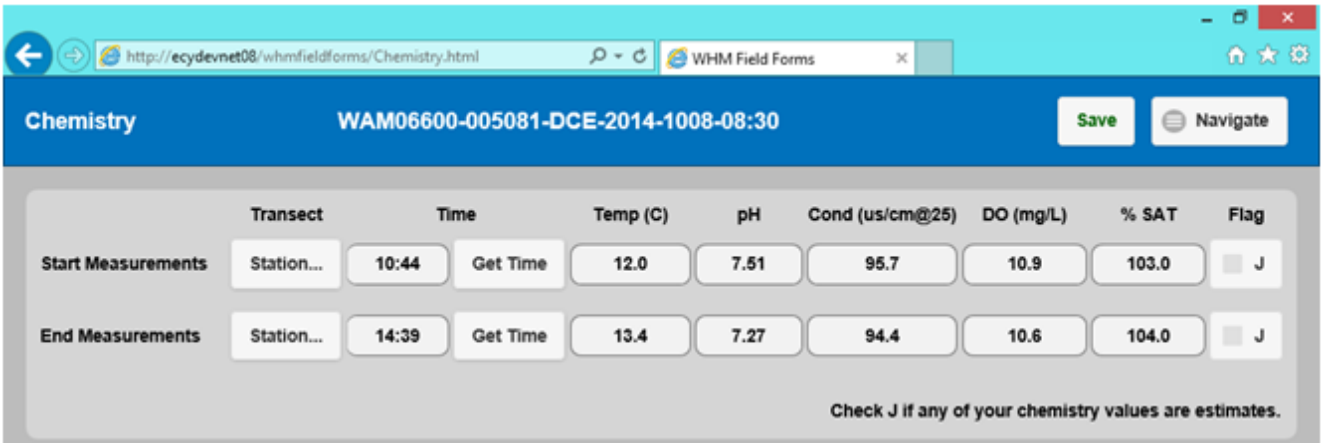

Figure 5. Top half of the chemistry page. Record measurements at the beginning and end of the DCE.

7.0 Records Management

7.1 Refer to SOP EAP125 (Janisch, 2017) which describes the process for validating, loading, and committing completed WHM electronic field forms to the WHM database.

8.0 Quality Control and Quality Assurance Section

8.1 Once monthly during July, August, September, and October, check the accuracy of the oxygen probe. Collect a Winkler sample as soon as possible following the initial calibration and prior to the first Data Collection Event. Subsequent Winkler samples should be collected immediately before and after monthly calibrations. For an accurate comparison, Winkler samples should be collected at the same station and time as the corresponding in situ DO reading. Winkler samples are collected and analyzed according to Ward and Mathieu (2013).

8.1.1 In situ DO readings are required to be within 1 mg/L of the average value from the paired Winkler samples. Verify that the meter measures to within 1 mg/L of the Winkler sample average, if it does not then recalibrate the probe and notify the project lead so that data entered from relevant prior sites can be recorded as suspect.

8.2 At the start and end of the field season, compare the measurements from the temperature probe against measurements of an NIST thermometer. Verify that the probe measures to within 1˚ C of the NIST thermometer in a cold water bath and to within 1˚ C of the NIST thermometer in a warm water bath. If it does not, then use a different probe that does meet these criteria.

8.3 QA/QC is discussed in the Quality Assurance Monitoring Plan (Cusimano et al, 2006), which is in the process of being updated.

9.0 Safety

9.1 All field staff must comply with the requirements of the EAP Safety Manual, especially Chapter 1 ‘General Field Work,’ which includes special circumstances like fall protection and working in rivers and streams. Sampling from a boat requires one person onboard to be a qualified boat operator and all persons onboard must be familiar with Chapter 3 of the EAP Safety Manual, ‘Boating.’

9.2 For further field health and safety measures refer to the EAP Safety Manual (Ecology, 2017).

10.0 References

10.1 Cusimano, R., G. Merritt, R. Plotnikoff, C. Wiseman, C. Smith, and WDFW. 2006. Status and Trends Monitoring for Watershed Health and Salmon Recovery: Quality Assurance Monitoring Plan. https://fortress.wa.gov/ecy/publications/SummaryPages/0603203.html

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10.4 Hach. 2010a. User Manual: pH Refillable Probe for Low Ionic Strength Samples: Model PHC28101 or PHC28103. Edition 1 (11/210). DOC022.53.80024. Hach Company, Loveland Colorado.

10.5 Hach. 2010b. User Manual: Luminescent Dissolved Oxygen Probe: Model LDO10101, LDO10103, LDO10105, LDO10110, LDO10115 or LDO10130. Edition 1 (11/210). DOC022.53.80021. Hach Company, Loveland Colorado.

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http://www.ecology.wa.gov/programs/eap/quality.html

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10.14 WOW. 2004. Water on the Web - Monitoring Minnesota Lakes on the Internet and Training Water Science Technicians for the Future - A National On-line Curriculum using Advanced Technologies and Real-Time Data. University of Minnesota-Duluth, Duluth, MN 55812. [Internet page, accessed 1-30-2017]
http://www.waterontheweb.org/under/waterquality/oxygen.html

11.0 Appendix A – Calibration Form

Hach HQ40D Calibration Form

Stream Name:_____ Site ID:_____

Date:_____ Time:_____ Operator:_____

Calibration: pH (daily), conductivity (daily), DO (monthly unless quality check fails).
Quality check: Before and after each DCE for pH, conductivity and DO.

Conductivity (SN:_____)
Standard used for calibration:_____µs/cm
Initial quality check[1]:_____ µs/cm
Final quality check[1]:_____ µs/cm
Calibration Successful: (Y/N)
Initial QA Successful?: (Y/N)
Final QA Successful?: (Y/N)

LDO (SN:_____)
Calibrate? (Y/N)
Initial quality check[6]:_____mg/L _____% Sat
Final quality check[6]:_____mg/L _____% Sat
Calibration Successful: (Y/N/Did not calibrate today)
Initial QA Successful?: (Y/N)
Final QA Successful?: (Y/N)

pH (SN:_____)
Percent Slope[2]:_____
Initial QC Reading[3]:_____ Initial QC Expected Value[4]_____
Final QC Reading[3]:_____ Final QC Expected Value[4]_____
Calibration Successful: (Y/N)
Initial QA Successful?: (Y/N)
Final QA Successful?: (Y/N)

pH Millivolts (Mv) [5]			
4	7	10	QC 7

Expected mV Range
pH4: 165 to 178 (<5)
pH7: -5 to +6 (<5)
pH10: -168 to +179 (<5)

Temp°C	Hach pH7	Hach pH10	NIST pH7
8	*	*	7.07
10	*	*	7.06
12	*	*	7.05
14	*	*	7.04
16	7.04	10.1	7.03
18	7.03	10.08	7.02
20	7.02	10.05	7.01
22	7.01	10.03	7.01
24	7	10.01	7
26	7	10	6.99

Comments

1: QA check with calibration standard. If meter conductivity is >± 10 µs/cm from standard, ***recalibrate or sample and flag data***

2: If <90%, buffers, probe, or cable may be bad

3: If meter pH is >± 0.10 units from Thermo pH 7 buffer (temp. corrected value) ***recalibrate or sample and flag data***

4: Value based on buffer temperature

5: Each probe will have a unique range for different buffers. By tracking this value over time, you can determine if a buffer is bad.

6: Measurement should be between 97.5 % and 102.5%. If not ***flag or recalibrate***

* Do not calibrate pH when buffers are below 15° C